UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
NATIONAL EXPOSURE RESEARCH LABORATORY
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OFFICE OF
RESEARCH AND DEVELOPMENT

November 28, 2018

Ken Kloo, Director
NJ Department of Environmental Protection
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Subject: NJ DEP Report #1: Targeted Analysis of PFCA in Soil Samples

Dear Mr. Kloo:

I am pleased to provide you with this initial laboratory report of targeted analysis results for perfluorinated carboxylic acid (PFCA) concentrations in soils. This is the first in a series of reports prepared as a part of EPA Office of Research and Development's (ORD) collaboration with the New Jersey Department of Environmental Protection (NJ DEP) and EPA Region 2 on the study, "Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Substances (PFAS) in Environmental Media from an Industrialized Area of New Jersey." This report includes concentration results for 13 PFCA in 24 soil samples. The ORD Principal Investigators (PIs) for this study are Drs. Andy Lindstrom, Mark Strynar, and John Washington. The results for this particular report were generated by Dr. John Washington in our Athens, Georgia laboratory. It is my understanding that these samples were collected by NJ DEP between October 23, 2017 and November 11, 2017 from various locations in the vicinity of the Solvay and Dupont facilities.

We do not interpret exposure or risk from the values presented in this report. EPA does not currently have health-based standards, toxicity factors, or associated risk levels for per- or poly-fluorinated alkyl substances (PFAS), other than perfluorooctanoic acid (PFOA), perfluorocatane sulfonate (PFOS), and perfluorobutanesulfonic acid (PFBS). While the data provided indicate the presence of certain PFCA in soil samples, it does not offer interpretation as to human or environmental exposure or risk.

Thank you for providing us with this opportunity for collaboration that helps to further both EPA's and New Jersey's understanding of an important public health issue. If you have any questions or concerns about this report, do not hesitate to contact me at (919) 541-2107 or via email at watkins.tim@epa.gov or Tim Buckley at (919) 541-2454 or via email at buckley.timothy@epa.gov. I look forward to our continued work together.

Sincerely,

Timothy H. Watkins
Director
National Exposure Research Laboratory
Office of Research and Development

Enclosure

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**Detection, Evaluation, and Assignment of PFAS in Environmental Media
from an Industrialized Area of New Jersey**

Laboratory Data Report #1: Targeted Analysis of PFCA in Soil

Background. EPA/ORD, EPA Region 2, and New Jersey Department of Environmental
Protection (NJ DEP) worked together to develop a study to evaluate sources, as well as the
nature and extent of PFAS contamination near manufacturing facilities in NJ. NJ DEP assumed
responsibility for the collection of samples and their shipment to ORD laboratories. ORD was
responsible for sample extraction and analysis. ORD personnel involved with laboratory analysis
and their roles and responsibilities are provided below in Table 1.

Table 1. EPA Office of Research and Development Analysis and Report Team.

Responsibility	Personnel
ORD Principal Investigators	Andy Lindstrom, Mark Strynar, and John Washington
Laboratory Chemistry	John Washington (PI) and Tom Jenkins
Quality Assurance Review	Brittany Stuart, Sania Tong-Argao
Management Coordination and Review	Brian Schumacher, Adam Biales, Myriam Medina-Vera, Tim Buckley
Report Preparation	Kate Sullivan, Tim Buckley

This report includes results for 24 soil samples and 4 duplicates and field blanks collected by NJ
DEP from November 8-10, 2017 and delivered to the ORD lab in Athens, GA on November 14,
2017. The results provided in this report were analyzed under the direction of Dr. John
Washington. This report does not contain results for 5 soil core samples collected at the same
time as analysis is not complete.

Thirteen perfluorinated carboxylic acid (PFCA) listed in Table 2 were analyzed with Ultra-
Performance Liquid Chromatography Mass Spectrometry (UPLC-MS) using methods described
within our Quality Assurance Project Plan (QAPP)[1] and that have been generally described in
Rankin et al., 2015[2]. These analytes were selected because previous reports have shown them to
be of concern. In brief, each sample was divided into three ~1 g aliquots. Each aliquot was
extracted with 90%/10% acetonitrile water followed by a liquid/liquid cleanup. Samples were
analyzed using a Waters Acquity UPLC coupled to a Waters Quattro Premier XE tandem mass
spectrometer. The reported concentrations are determined as the mean value of triplicate aliquot
analysis (Table 3). PFCA concentrations were determined using mass-labeled internal calibration
curves for quantitation using a traditional targeted analysis approach. These analyses were
performed on samples, process blanks, and check standards. Dilution of samples was not
performed.

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[1] National Exposure Research Laboratory, Quality Assurance Project Plan: Detection, Evaluation and Assignment of
Multiple Poly and Per-fluoroalkyl Substances (PFAS) in environmental media from an industrialized area of New
Jersey. Prepared for New Jersey Department of Environmental Protection (NJ DEP), September 14, 2017.

[2] K. Rankin, S. A. Maybury, T.M. Jenkins, J.W. Washington, A North American and global survey of perfluoroalkyl
substances in surface soils: Distribution patterns and mode of occurrence. Chemosphere 161, 333-341 (2015).

Table 2. PFCA Analyzed in NJ Soil Samples by UPLC-MS.

Acronym	Chemical Name	Formula	CAS no.	Monoisotopic Mass (g/mol)
PFBA	Perfluorobutanoic Acid	$C_4HF_7O_2$	375-22-4	213.9865
PFPeA	Perfluoropentanoic Acid	$C_5HF_9O_2$	2706-90-3	263.9833
PFHxA	Perfluorohexanoic Acid	$C_6HF_{11}O_2$	307-24-4	313.9801
PFHpA	Perfluoroheptanoic Acid	$C_7HF_{13}O_2$	375-85-9	363.9769
PFOA	Perfluorooctanoic Acid	$C_8HF_{15}O_2$	335-67-1	413.9737
PFNA	Perfluorononanoic Acid	$C_9HF_{17}O_2$	375-95-1	463.9705
PFDA	Perfluorodecanoic Acid	$C_{10}HF_{19}O_2$	335-76-2	513.9673
PFUnDA	Perfluoroundecanoic Acid	$C_{11}HF_{21}O_2$	2058-94-8	563.9641
PRDoDA	Perfluorododecanoic Acid	$C_{12}HF_{23}O_2$	307-55-1	613.9609
PFTrDA	Perfluorotridecanoic Acid	$C_{13}HF_{25}O_2$	72629-94-8	663.9577
PFTeDA	Perfluorotetradecanoic Acid	$C_{14}HF_{27}O_2$	376-06-7	713.9545
PFHxDA	Perfluorohexadecanoic Acid	$C_{16}HF_{31}O_2$	67905-19-5	813.9482
PFODA	Perfluorooctadecanoic Acid	$C_{18}HF_{35}O_2$	16517-11-6	913.9418

Quality Assurance

Data were checked for compliance with a number of laboratory and field related quality control evaluation criteria as specified in the project QAPP. Quality control results indicated that analyses were generally within expected performance criteria. Some individual analyte/sample values failed criteria and are flagged accordingly in Table 3. Detailed QC results are provided in Appendix A, including an explanation of data rating criteria used in the results Table 3. Several field blank analytes were found in low concentrations > LOD.

Results

Concentration results for 24 soil samples identified by sample IDs assigned by NJ DEP are presented in Table 3 along with 2 field duplicates and 2 field blanks. Results are reported for 13 PFCAs that range from C4 through C18. Soil sample and duplicate concentrations are reported as the mean of three sample aliquots in units of mass of PFCA per unit mass of dry solid, i.e., pg/g, Field blank concentrations represent one aliquot only.

- Concentrations across all analytes and samples ranged from <LOD to values that exceeded our calibration curve. In general, lower concentrations were observed for C4-C8, C16, and C18 based on the number of values that were either <LOD or <LOQ. Across all samples, the highest concentrations were observed for C11 where two samples (PFSS001 and PFSS008) exceeded our calibration curve with estimated concentrations of 5,730 and 6,560 pg/g, respectively. These values are flagged accordingly with "JC1".

- Mid-carbon length compounds (PFOA, PFNA, PFDA, and PFUA) were consistently present in higher concentrations than other compounds, although concentrations were variable from site to site. The odd-chain lengths C9, C11, and C13 exceeded the one-carbon homologues, C8, C10 and C12, in numerous samples, a pattern that is unusual in our experience.

Table 3. PFCA Concentrations (pg/g) in Soil Samples Determined with Targeted Analysis.

Carbon Length	C4	C5	C6	C7	C8	C9	C10	C11	C12	C13	C14	C16	C18
NJDEP Sample ID	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUA	PFDoA	PFTrA	PFTeA	PFHxDA	PFODA
PFSS001	121 (U)	143	ND	91.4 (U)	398	2710	381	5730 (JC1)	99.4	590	24.7	ND	ND
PFSS002	102 (U)	52.2 (U)	ND	31.1 (U)	196 (JP)	91.2	115	1480	64.7	153	24.9	ND	ND
PFSS003	149	153	ND	159	422	770	581	1440.	244	252	104	ND	ND
PFSS004	ND	366 (U)	296 (U)	345	1070 (U)	2630 (JP)	625 (U)	2770 (JP)	183 (U)	326 (JP)	81.6 (U)	ND	ND
PFSS005	112 (U)	178	ND	132	244 (U)	691	231	825	60.9	85.6	28.0	ND	ND
PFSS006	119 (U)	245	ND	102	295 (U)	766	278	1370	111	165	46.5	ND	ND
PFSS007	257	276	186 (U)	188	749	1220	256	810	63.3	91.6	39.9	ND	ND
PFSS008	69.4 (U)	53.3	ND	37.7 (U)	ND	295	324	6560 (JC1)	277	1280	71.5	ND	ND
PFSS009	103	104	ND	91.2	162 (U)	522	207	1460	84.7	226	35.4	ND	ND
PFSS010	ND	77.7	158 (U)	151	1900	286	262	794	100	104	39.8	ND	ND
PFSS011	192	176 (U)	155 (U)	100	317	784	422	1370	117	166	54.9	ND	ND
PFSS012	88.8 (U)	171	ND	130	573	445	268	655	120	97.4	47.5	ND	ND
PFSS013	159	199	426 (U)	135	619	1150	312	1250	146	187	69.7	ND	ND
PFSS014	228 (JP)	192	228 (U)	139	415 (JP)	887 (JP)	337	803	103	97.9	36.9	ND	ND
PFSS015	153 (U)	213 (JP)	255 (U)	163 (JP)	599 (JP)	614 (JP)	473 (JP)	3970 (JP)	145 (JP)	347 (JP)	50.5 (JP)	ND	ND
PFSS016	91.4	213	268 (U)	172	264	651	296	307	74.1	54.3	31.5	ND	ND
PFSS017	141	295	320	319	405	711	1460	1160	448	134	114	39.0 (U)	ND
PFSS018	119 (U)	96.1	ND	66.3	160 (U)	356	364	377	113	65.1	45.0	ND	ND
PFSS019	143 (U)	150 (U)	ND	68.3 (U)	151 (U)	337 (U)	193	543	83.8	78.7	38.0	ND	ND
PFSS020	65.1 (U)	121	ND	56.4	92.7 (U)	475	192	1340	54.6	112	19.1	ND	ND
PFSS021	133 (U)	247	ND	176	689	626	401	895	150	129	71.2	ND	ND
PFSS022	760	622	ND	292	1250	658	697	1020	447	300	257	75.9 (U)	86.5 (JP)
PFSS023	283 (JP)	381	226 (U)	446	330 (U)	446	1060	1080	525	203	251	106 (U)	30.8 (U)
PFSS024	257	425	493 (U)	426	892	731	755	844	199	87.3	70.9	ND	ND
PFSSDUP1	152 (U)	535	526 (U)	509	804	862	765	858	193	90.5	70.8	ND	ND
PFSSDUP3	166	334	ND	94.2	185 (U)	846	241	1080	84.9	122	42.0	ND	ND
PFSSFB1	6.88 (U)	6.96 (U)	ND	ND	ND	7.94	ND	ND	1.81	ND	1.04 (U)	ND	ND
PFSSFB2	ND	ND	ND	7.52 (U)	ND	10.1	ND	1.25 (U)	13.7	ND	6.37	ND	ND

ND: Sample result is less than the limit of detection (<LOD).

U: Sample result is less than the limit of quantitation (<LOQ). The value is an estimate.

JP: Sample triplicates do not meet acceptance criteria for precision. The value is an estimate.

JC1: Sample result exceeds the upper calibration range. The value is an estimate.

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Appendix A

Quality Assurance Documentation

The quality assurance and control analyses described in this Appendix A refer to 24 soil samples (labeled PFSS) received at ORD's Athens Georgia laboratory on November 14, 2017. The soil samples included 2 field duplicates and 2 field blanks. Soil core samples labeled PFSC (n=4 plus one duplicate) were also received on this date. Analysis of soil core samples has not been completed.

All samples (excluding field blanks) were extracted, divided into three ~1g aliquots, and analyzed in triplicate. Targeted analysis results presented in Table 3 represent the average of 3 aliquots. The Limit of detection (LOD) and Limit of Quantitation (LOQ) were determined for each analyte based on statistical comparison with the process blanks with 95% confidence.

Quality control assessment includes measures for accuracy based on internal standard recovery and calibration standard checks. Bias is determined by instrument and field blanks. Precision is determined for analytical and field methods. The QAPP for this project specifies measured objectives. Table A1 lists the quality measures, quality objectives and summarizes results. Data not meeting certain criteria are flagged in the results Table 1 as defined in Table A1.

In summary:

- We observed excellent recoveries of internal standards in both samples (99 ± 9%) and process blanks (97 ± 18%) where ± indicates the relative standard deviation.

- Check standards of varying concentrations spanning the calibration range (n=7) were analyzed in conjunction with samples to monitor instrument performance. The mean recovery across the 13 analytes and 7 standards (n=91) was 95.7% ranging from 0 to 176%.

- Concentration estimates for C16 and C18 are more uncertain than C4-C14 congeners due to failure of some check standards and additional analytical difficulties. These congeners are flagged for all samples accordingly.

- Analytical precision was indicated by repeated analysis of two samples. All detected analytes met analytical QC criteria of CV ± 50%.

- For samples with average analyte concentrations > LOQ, the CV was ≤ 50% in 18 of 24 samples (75%). Results that did not meet QC criteria (CV > 50%) are flagged accordingly with "JP" in Table 3.

- Duplicate samples are compared assuming that PFSSDUP1 was collected at site PFSS024 and that PFSSDUP3 was collected at site PFSS006. The mean CV for analytes >LOQ was 19.1% ranging from 1 to 64%.

- Several analytes were detected in the field blanks above the LOD. The samples labeled as "Field Blanks," are more appropriately considered "Field Controls." PFCA concentrations were observed >LOQ for C9, C12, and C14. We attribute these trace levels to contamination of the original Ottawa sand rather than field or laboratory contamination. Therefore, no blank adjustment was made for reported sample concentrations.

Table A1. Quality Assurance Evaluation Results

Data Quality Indicator	Quality Control Measure	Quality Objective	Summary of Results	Corrective Action Taken
Accuracy	Internal standard recovery ($^{13}C_8$-PFOA)	± 50%	All results within acceptance limits Samples: 99% ± CV 10% Process Blanks: 97 ± CV 19%	None necessary

	Calibration check standards: 7 standards x 13 analytes (n=91)	± 50%	• Mean recovery was 95.7%
			• 6 out of 91 exceedances:
			   ○ 5 occurred at level of lowest calibration standard and not expected to affect results
			   ○ 1 occurred a level of second lowest calibration standard
				• Sample results for C5, C6, and C9 are at a level greater than that of the associated failed check standard. Exceedances are not anticipated to significantly impact sample results.
				• Results for C16 and C18 are estimated and flagged in results table.

Bias	Instrument blanks	<LOD	All instrument blanks were free of reported analytes.	None necessary
	Field blanks (n=2)	None	• C9, C12, and C14 detected above the LOQ	
Precision*	Analytical precision Repeated extraction of a homogenized sample (n=2)	CV ± 50%	Of 21 sample/analyte comparisons for C4 to C14 >LOD: • All analytes > LOQ met quality objective. • CV was ± 8.0% (range 0 to 30%)	None necessary
	Sample precision	CV ± 50%	• 18 out of 24 samples with results >LOQ met criteria	Results exceeding criteria are flagged with "JP"
	Field duplicates (n=2)	CV ± 50%	Mean CV 17% (range 0.8 to 58.6%) -1 analyte >LOQ exceeded criteria	Results exceeding criteria flagged as "JP"
Method sensitivity	Limit of detection (LOD)	None specified	Defined as the level at which the sample results statistically exceed the level in the process blanks with 95% confidence	Results <LOD or with no detectable peak flagged with "ND"
	Limit of quantitation (LOQ)	None specified	Defined as the level at which the sample results statistically exceed the level in the process blanks with 99.9% confidence.	Results <LOQ flagged with "U" Result >LOQ are reported as blank corrected.

*Quality objective for precision measurements was applied to results > LOQ. Results that are <LOQ are already flagged as estimates with "U".